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EXPERIMENTAL AND THEORETICAL STUDY IN RUBBER REINFORCED WITH CARBON FILLERS UNDER TENSION–CYCLIC LOAD

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ABSTRACT

The main goal of this study is the selection of the best rubber reinforced with carbon black fillers stock to resist the tension, tear and crack growth due to cyclic stresses. The mechanical properties of this rubber is determined, the test specimens are prepared and manufactured in Babylon Tires Factory locate in Al-Najaf Al-Ashraf city in Iraq. In this work, the objectives are achieved experimentally about 80% and theoretically about 20%, the experimental works consisting tear and cyclic crack growth tests to find the crack growth length with number of cycles. The theoretical analysis is applied in special Paris law equations to calculate the crack growth length with number of cycle. The comparison between experimental and theoretical results of crack growth are determined and it is found that the maximum discrepancy in the results is about 14.28%.

KEYWORDS: Reinforced Rubber, Carbon Black Fillers

INTRODUCTION

The rubbers are widely used as an insulating material for electrical wires, cables, ropes, coatings, transformers, motor winding, tires etc. The natural rubber is obtained from rubber tree hevea brasiliensis. The natural rubber has a limited applications, because of its resistance at high temperatures, Mars, and, Fatemi, 2003.

There are many studies that discuss the rubber recipes contents and the function of all elements, and their effects on mechanical properties (such as tensile strength, tear strength, fatigue resistant…etc.).

J. L. Koenig, 1999, studied and discussed the vulcanization mechanism for chemical reactions of network structures in elastomers.

S. S. Choi, 2000, found that the properties of the carbon black filled NR compounds and their vulcanizes were found to be different with the mixing procedure although they had the same formulation.

S. Chuyjuljit et al, 2002, investigated the effects of particle size and amount of two types of fillers, namely, carbon black and calcium carbonate, on the curing characteristics and dynamic mechanical properties of vulcanized natural rubber.

J. H. Kim, and H. Y. Jeong, 2005, studied the material properties and fatigue life of natural rubber compounds filled with different carbon blacks N330, N650 and N990. The fatigue life was obtained by conducting a displacement-controlled fatigue test on an hour glass-shaped specimen.

A. Robisson, 2010, analyzed carbon black filled elastomers as random three – dimensional cellular solids whereas bending arms are made of carbon black clusters with bound rubber, and where the matrix was the free rubber. Both phases are co-continuous.

A. D. Drozdov and N. Dusunceli, 2014, reported on carbon black-filled thermoplastic elastomeric in multi-step uniaxial tensile cyclic tests with various strain rates at room temperature. Experimental data reveal several unusual features of stress-strain diagrams. Adjustable parameters in the stress-strain relations are found by fitting the experimental data. Ability of the constitutive equations to describe the mechanical behavior of thermoplastic-elastomeric composite under cyclic deformation.

THEORETICAL ANALYSIS

The mechanical properties of rubbers are derived from the molecular chain that is assembled into a cross-linked network. When unstressed, the molecular segments between crosslinks are randomly coiled. When a tensile stress is applied, deformation is principally achieved by localized motions of the molecular segments that take on new shapes, in which they are stretched out in the direction of the tensile axis, De, and, White, 2001.

Fatigue Crack Growth Behavior

The crack growth approach explicitly considers preexisting cracks or flaws. The idea of focusing attention on individual flaws was introduced by Inglis, 1913 and Griffith, 1920. Griffith proposed a fracture criterion based on an energy balance including both the mechanical energy of a cracked body, and the energy associated with the crack surfaces.

An alternate analysis for fatigue crack growth is based on stress intensity factor (SIF) and involves the use of Paris law. While the Paris law is widely accepted in classical fracture mechanics, its application to elastomeric materials has been debated. However, it may shed light on the mechanics of failure in this analysis. For an applied load that is cycled uniformly between K_{max} and K_{min}, the Paris Law relation is given by, Schuble et al,2004, and Gdoutos,1993.

$$da/dN=A(\Delta K)^{\alpha} \tag{1}$$

where A and α are material constants, and,

$$\Delta K=K_{max} - K_{min} \tag{2}$$

where ΔK is the stress intensity factor range. The fracture occurs for different shapes of test piece and under varied loading conditions at a characteristic value of a “critical stress intensity factor” K_c, defined as, Mark et al, 2005.

$$K_c=Y*\sigma_c*\sqrt{(\pi*a_c)} \tag{3}$$

where K_c is the fracture toughness, a property that is a measure of a material’s resistance to brittle fracture when a crack is present. Y is a dimensionless parameter or function that depends on both crack and specimen sizes and geometries, as well as the manner of load application. Relative to this Y parameter, for planar specimens containing cracks that are much shorter than the specimen width, has a value of approximately unity.

Prediction of Fatigue Life

The prediction of fatigue life in elastomer materials involves the application of a fracture mechanics method on simple geometries. It was proved that fatigue crack growth can be predicted by the application of fracture mechanics with a

high degree of accuracy, and could prove to be a good tool for fatigue crack growth problems since it deals with material properties rather than geometric properties and loading conditions. The fatigue life of the material can be calculated using the following equation, Liu, 2011,

$$N_f = \int_{a_o}^{a_c} \frac{da}{A(\Delta K)^a} \tag{4}$$

Where, a_o and a_c are the initial and final crack length respectively.

EXPERIMENTAL WORKS

The mechanical and physical properties under some influential conditions are discussed, including (NR, SBR, BRcis) blending and different types of carbon black to produce the flexibility for rubber compound. Understanding this advantage of adding different types of carbon black and its resulting effect on the mechanical behavior also, hence the final product was the main motivation to do further test to improve the existing materials used in Babylon Tires Factory. Some of these testes were carried out in the factory labs.

Blend Materials

Table 1 shows the types of blends under study. The blends manufacturing processes are summarized in the following steps:

- Putting a raw of gum of elastomers in the gap between the two mill rolls, at a mill roll opening of 0.2 cm at 70oC for (2 minutes) as shown in Figure 1.
- The process is repeated many times until the material is well calendared. The speeds of the two rolls are often different, the back roll rotating faster than the front roll. See laboratory calendar machine as shown in Figure 2a.
- Add stearic acid for (2 minutes).
- Add other ingredients, such the zinc oxide, one-half of black and add process dutrex oil for (4 minutes) as shown in Figure 2b.
- Add remainder of the black for (2 minutes) as shown in Figure 2c.
- Cooling the master batch to room temperature.
- Add Sulfur to the master batch stock for (1 minute).
- Add the accelerator (CBS) for (1 minute).
- Banding the master batch stock with the mill opening at 0.3 to 0.1 cm for (1minute) as shown in Figure 2d.
- Sheeting the batch to minimum thickness of 0.6 cm for (5 minutes).
- Cooling the batch to room temperature. Total time is about (30 minutes).

Molds Preparation

Two different molds are used in this work. One of them has a cavity with the dimensions of (160*160*6 mm^3) from which test samples are cut by using cutting tool and hydraulic press, such as tensile, hardness, specific gravity, thermal conductivity, thermal diffusivity and resilience test specimens. The second one has a cavity with (160*100*10

mm^3), from which swelling and abrasion test specimens are cut. Before compression molding and vulcanization take place, mold must be cleaned and lubricated by oil to facilitate product releasing from mold later.

Vulcanization Procedure

The process of rubber vulcanization is used and summarized in the following steps:

- Heating the two molds by an electric source from (30 to 200±1°C).
- The empty mold brought to curing temperature within (±1°C) in the closed press, and held at this temperature for at least 20 minutes before the unvulcanized pieces are inserted. The temperature of the mold is verified by a thermocouple inserted in one of the overflow grooves and in intimate contact with the mold.
- The press is opened Figure 3, then the unvulcanized (uncured) sheets are inserted into the mold, and the press is closed as soon as possible. When the mold is removed from the press to insert the sheets, the precautions must be taken to prevent excessive cooling of the mold by contact with cool metal surfaces or by exposure to air drafts.

Cooling and Cutting Process

The cooling is the process after releasing the thin sheet and the specimen from the mold; they are left to cool down in a room temperature for at least eight hours to get the thin sheet of rubber. Finally, by using the cutter (Wallace) shown in Figure 4, the standered dumbbell specimens were obtained under ASTM D412 specifications for tensile and tension-compression cyclic loading, as shown in Figure 5.

All above steps were repeated to produce the other blends used in this work.

Tear Test

Tear resistance in rubber may be described as the resistance to growth of a nick or cut when tension is applied to the tear specimen, Winspear, 1968.

The cutting of test pieces shall be obtained from the molded test specimen sheets. The cutting die was used to cut the test pieces from the sheet with a single impact stroke to ensure smooth cut surfaces. A razor-nicked test piece with a crescent shape and with tab ends was used, type B from ASTM D 624, the applied force on the test piece acts in a direction substantially along the major axis (length) as shown in Figure 6 and perpendicular to the “nick”, or razor cut. The nicking device shown in Figure 7 secures the test piece in a manner that prevents any movement; so that the cutting mechanism introduces a razor blade on a plane perpendicular to the major axis of the test piece to make a nick to a depth of 0.50+0.05 mm.

A tearing strain is applied to the test specimen by means of a tensile testing machine operated without interruption at a constant rate until the specimen is completely torn. This test method measures the force per unit thickness required to rupture, initiate, or propagate a tear through a sheet of rubber in the form of one of several test piece geometries. The testing machine shall conform to the requirements as specified in test method described by ASTM D 412. The rate of jaw separation shall be 500 mm/min.

Cyclic Fatigue and Crack Growth Tests

The most important fatigue failure is called flex cracking. The causes of this failure are, stress breaking of rubber

chains and cross-links and, more important, oxidation accelerated by heat build up due to cyclic loading. This test method covers the determination of crack growth of vulcanized rubber when subjected to cyclic loading. It is particularly applicable to tests of synthetic rubber compounds which resist the initiation of cracking due to cyclic loading when tested by test metric AX- M500-25kN as shown in the Figure 8.

These uniaxial cyclic tests are conducted on dumbbell specimens shown in Figure 9 under controlled strain, for different speed tests, three loading cycles at constant strain level were carried out for all kinds of blends. Each test can be managed by using a computer connected to the machine which fully controls the test using win test analysis universal testing software. This is a multi-functional and fully customizable software package that supports all industry standards including ISO, ASTM and BS EN specifications. Test specifications supported include tensile, compression, flexural, peel, tear, burst, adhesion, shear, cyclic and hardness. Additional flexibility is provided by user-defined multistage step testing for highly specialized testing requirements. The input data to the program to perform the test are specimen length, width, thickness, strain level and speed test.

RESULTS AND DISCUSSIONS

According to the tests of tensile strength, modulus of elasticity, elongation at the breaking point, tearing strength and the value of hardness, the optimum stock design was the stock composition shown in Table 1,which is used later to find the best quantity and type of carbon black filler type additive to the stock. The stocks with carbon black filler are shown in Table 2. The number of cycles versus crack growth length experimentally and theoretically are listed and plotted in Table 3 and curve Figure 10 respectively, these results are estimated according to cyclic tension – compression test experimentally as shown in Figure 9, and are calculated according to Paris law equations as listed from eq. (1) to eq. (4) theoretically. The experimental cyclic test specimen must be conditioned at least 12 hr. at the test temperature. At least three specimens from sample shall be tested and the results averaged. Since the rate of crack growth is important, take frequent readings early in the test observe the specimens, and measure the length of the developed crack. Use of an electronic metric scale for measuring crack growth is recommended if available. The comparison between the experimental and the theoretical crack growth results shows that the maximum percentage of discrepancy is 14.28%.

Table 1: The Best Composite Stock According to the Mechanical Properties Tests

Master Batch				
No	**Material**	**(pphr)**	**Percentage (%)**	**Weight**
1	NR	50	28.94	100
2	SBR	30	17.37	60
3	BR	20	11.58	40
4	C.B. 550	51	29.52	102
5	St. Acid	2	1.16	4
6	Zinc Oxide	4	2.31	8
7	Oil (Dutrex)	8.5	4.92	17
8	IPPD (Anti-Ozonant)	1.75	1.01	3.5
9	TMQ (Anti-Oxidant)	1	0.58	2
10	Wax (Paraffin)	2	1.16	4
	Batch			
8	CBS	0.5	0.29	1
9	Sulfur	2	1.16	4
	SUM	172.75	100(%)	345.5

Table 2: The Stocks after Adding Carbon Black Filler

Stock No.	Carbon Black Type and Percentage pphr		
	N330	N550	N660
1	51	-	-
2	25.5	51	-
3	-	-	51
4	-	25.5	25.5

Table 3: Experimental and Theoretical Crack Growth Cyclic Test Results of Stock No.1

Number of Cycles	Experimental Crack Growth Length (mm)	Theoretical Crack Growth Length (mm)	Percentage of Discrepancy (%)
1253	2.5	2.875	13%
2744	3.5	3.944	11.25%
4988	3.9	4.123	5.4%
6642	4	4.351	8.06%
8340	4.2	4.9	14.28%
10465	4.9	5.121	4.31%
12790	5.2	5.623	7.52%
14388	5.9	6.377	7.48%

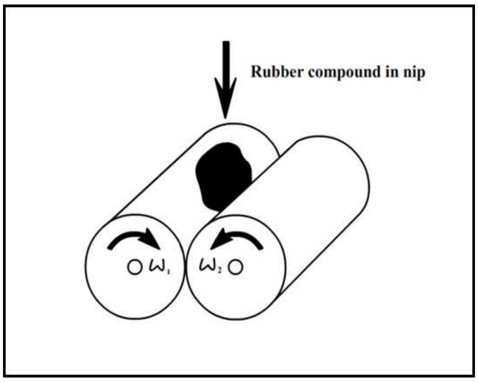

Figure 1: Two Roll Mill

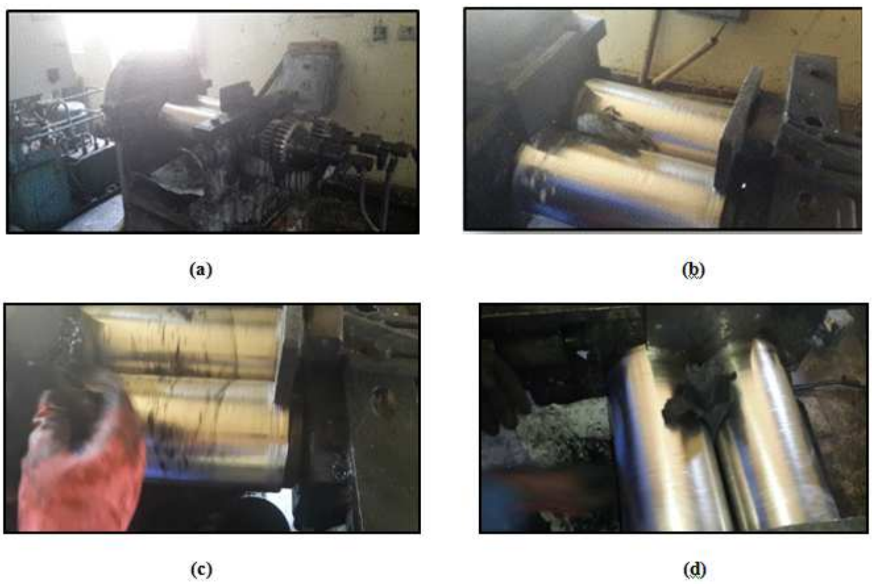

Figure 2: Blends Manufacturing Process

Figure 3: Thermal Hydraulic Press

Figure 4: Wallace Sample Press

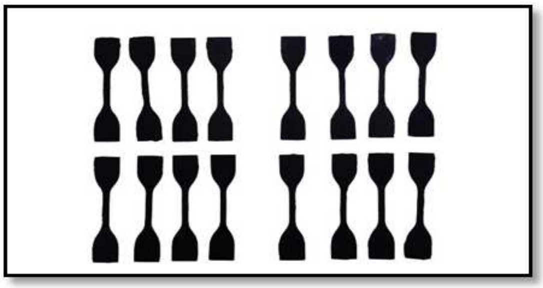

Figure 5: Dumbbell Test Specimens (Tension-Compression Cyclic Specimens)

Figure 6: Tear Specimen

Figure 7: Nicking Device

Figure 8: Cyclic Tension-Compression Test Machine

Figure 9: Crack Growth Cyclic Steps of Sample Stock under Testing

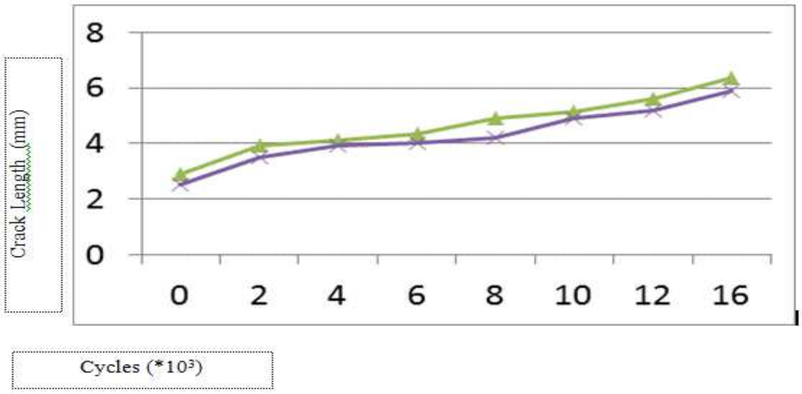

Figure 10: The Number of Cycle versus Crack Growth Length, Experimentally and Theoretically

CONCLUSIONS

The following are the main summarized conclusions raised by this study:

- In general, the tensile strength of NR/BR/SBR blends shows an increasing trend with an increase in the carbon black addition.
- The elongation at the breaking point of the blends decreases with the increasing of carbon black addition.
- The crack growth is increased proportionally with the increasing of the percentage of carbon black, and with increasing the number of cycles.
- The comparison of results between the experimental and the theoretical estimation of crack growth are determined to have the value of maximum discrepancy of 14.28%.

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